AFRL-RZ-WP-TP-2012-0135

TEXTURED ION-BEAM ASSISTED DEPOSITION: MAGNESIUM OXIDE TEMPLATE ON NON-METALLIC FLEXIBLE CERAFLEX FOR EPITAXIAL GROWTH OF PEROVSKITE FILMS (POSTPRINT)

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FEBRUARY 2012

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REPORT DOCUMENTATION PAGE

Form Approved OMB No. 0704-0188

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1. REPORT DATE *(DD-MM-YY)*	2. REPORT TYPE	3. DATES COVERED *(From - To)*
February 2012	Journal Article Postprint	04 April 2005 – 04 April 2007

4. TITLE AND SUBTITLE
TEXTURED ION-BEAM ASSISTED DEPOSITION: MAGNESIUM OXIDE TEMPLATE ON NON-METALLIC FLEXIBLE CERAFLEX FOR EPITAXIAL GROWTH OF PEROVSKITE FILMS (POSTPRINT)

5a. CONTRACT NUMBER In-house

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER 62203F

6. AUTHOR(S)
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5d. PROJECT NUMBER 3145

5e. TASK NUMBER 32

5f. WORK UNIT NUMBER 314532ZE

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

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Air Force Materiel Command, United States Air Force

8. PERFORMING ORGANIZATION REPORT NUMBER AFRL-RZ-WP-TP-2012-0135

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
Air Force Research Laboratory
Propulsion Directorate
Wright-Patterson Air Force Base, OH 45433-7251
Air Force Materiel Command
United States Air Force

10. SPONSORING/MONITORING AGENCY ACRONYM(S) AFRL/RZPG

11. SPONSORING/MONITORING AGENCY REPORT NUMBER(S) AFRL-RZ-WP-TP-2012-0135

12. DISTRIBUTION/AVAILABILITY STATEMENT
Approved for public release; distribution unlimited.

13. SUPPLEMENTARY NOTES
Journal article published in the *Journal of Electronic Materials*, Vol. 36, No. 10, 2007. © 2007 TMS. The U.S. Government is joint author of the work and has the right to use, modify, reproduce, release, perform, display, or disclose the work.
PA Case Number: AFRL/WS-07-0633; Clearance Date: 04 Apr 2007. Work on this effort was completed in 2007. Paper has color content.

14. ABSTRACT
This paper reports on a systematic study of the development of bi-axially textured magnesium oxide (MgO) templates on flexible ceramic polycrystalline ceraflex substrates by ion-beam assisted deposition (IBAD) and the preliminary test of the growth of $YBa_2Cu_3O_7$ on top. The rough surface of the original ceraflex, with a typical roughness of about 100 nm, presented a serious challenge in the development of textured IBAD-MgO. By a coating of multiple layers of spin-on-glass (SOG), the surface roughness has been reduced to about a few nanometers. After an amorphous Y_2O_3 buffer had been deposited on multilayer SOG coated ceraflex, IBAD-MgO and homo-epitaxial MgO were grown with good in-plane texture of $\Delta\phi \sim 9.3°$ An epitaxial $SrTiO_3$ film was subsequently deposited on this textured template with in-plane $\Delta\phi \sim 10.8°$. Finally, an epitaxial $YBa_2Cu_3O_7$ superconducting film was grown on $LaMnO_3$ buffered and IBAD-MgO textured ceraflex, and AC susceptibility examination indicated the transition temperature was 88 K. The results have demonstrated that an IBAD-MgO textured template on ceraflex can be used for the epitaxial growth of perovskite films.

15. SUBJECT TERMS
systematic study, ceraflex, nanometers, ion-beam assisted deposition, preliminary test, ceramic polycrystalline, epitaxial, film

16. SECURITY CLASSIFICATION OF: a. REPORT	b. ABSTRACT	c. THIS PAGE	17. LIMITATION OF ABSTRACT:	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON (Monitor)
Unclassified	Unclassified	Unclassified	SAR	14	Timothy J. Haugan **19b. TELEPHONE NUMBER** *(Include Area Code)* N/A

Standard Form 298 (Rev. 8-98)
Prescribed by ANSI Std. Z39-18

Journal of ELECTRONIC MATERIALS, Vol. 36, No. 10, 2007
DOI: 10.1007/s11664-007-0196-3

Special Issue Paper

Textured Ion-Beam Assisted Deposition: Magnesium Oxide Template on Non-Metallic Flexible Ceraflex for Epitaxial Growth of Perovskite Films

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This paper reports on a systematic study of the development of bi-axially textured magnesium oxide (MgO) templates on flexible ceramic polycrystalline ceraflex substrates by ion-beam assisted deposition (IBAD) and the preliminary test of the growth of $YBa_2Cu_3O_7$ on top. The rough surface of the original ceraflex, with a typical roughness of about 100 nm, presented a serious challenge in the development of textured IBAD-MgO. By a coating of multiple layers of spin-on-glass (SOG), the surface roughness has been reduced to about a few nanometers. After an amorphous Y_2O_3 buffer had been deposited on multilayer SOG coated ceraflex, IBAD-MgO and homo-epitaxial MgO were grown with good in-plane texture of $\Delta\phi \sim 9.3°$ An epitaxial $SrTiO_3$ film was subsequently deposited on this textured template with in-plane $\Delta\phi \sim 10.8°$ Finally, an epitaxial $YBa_2Cu_3O_7$ superconducting film was grown on $LaMnO_3$ buffered and IBAD-MgO textured ceraflex, and AC susceptibility examination indicated the transition temperature was 88 K. The results have demonstrated that an IBAD-MgO textured template on ceraflex can be used for the epitaxial growth of perovskite films.

Key words: IBAD-MgO, textured template, ceraflex, perovskite films

INTRODUCTION

Perovskite films are widely used for many different functional devices, including high temperature superconductors (HTSs), ferro-electric devices, *etc.* Among these applications, HTS $YBa_2Ca_3O_7$ (YBCO) coated conductors have been very technologically attractive in the past several years. During the fabrication process, highly textured perovskite buffer layers, such as $SrTiO_3$,[1] $SrRuO_3$,[2] and $LaMnO_3$,[3] were usually used to provide improved structure and chemical compatibility for the epitaxial growth of YBCO. These perovskite ABO_3 buffers were also used for the hetero-epitaxial growth of ferro-electric films.[4] So far, high current capacity YBCO coated conductors have been demonstrated on flexible metallic tapes.[5] However, AC losses, which are induced in the superconducting layer and metallic substrate by an exterior alternating electro-magnetic field, can be of serious concern in the application of YBCO coated conductors.[6,7] The eddy current loss in the substrate, which can be substantial at higher frequencies,[8] can be avoided if the metallic substrate can be replaced with a non-metallic substrate.

A novel flexible ceramic substrate, ceraflex,[9] is a potential substrate for many devices, including a more AC-tolerant HTS coated conductor in some applications, and its uses are still being explored. Table I[9] briefly shows the properties provided by the manufacturer. Ceraflex is yttria stabilized zirconia (YSZ), which has a resistivity on the order of a few hundred ohm centimeters, 6–8 orders of magnitude higher than that of the metallic substrates currently

(Received November 20, 2006; accepted May 22, 2007; published online August 7, 2007)

Table I. Properties of Ceraflex

Product Description	3 mole% yttria	8 mole% yttria
Formula	ZrO_2 with 3 mole% yttria	ZrO_2 with 8 mole% yttria
Resistivity (600°C)	280 ohm cm	120 ohm cm
Radius of curvature	8 mm	–
Bending strength (@ 25°C)	120 kgf/mm^2	40 kgf/mm^2
Standard thickness	0.1 mm and 0.2 mm	0.1 mm and 0.2 mm
Density	6.00 g/cm^3	5.85 g/cm^3
Surface roughness (R_a)	approx. 100 nm	approx. 100 nm
Thermal expansion coefficient (25–800°C)	9.9×10^{-6}/°C	9.5×10^{-6}/°C
Thermal conductivity (@ 25°C)	7.6×10^{-3} cal/cm s °C	6.7×10^{-3} cal/cm s °C

employed for HTS coated conductors.[10] Ceraflex has two different stoichiometries, which have comparable properties except for their flexibility. However, even if it is chemically compatible with YBCO growth, it is polycrystalline and cannot provide the in-plane textured surface for YBCO epitaxy. It is well known that YBCO is highly anisotropic, with preferred electron transport in the Cu-O planes (ab-plane). In addition, the d-symmetry of the electron pairing in HTS has posted a strict requirement for the in-plane alignment. Exponential decrease of critical current density in YBCO with both in-plane and out-of-plane misorientations has been observed, and the critical misorientation angles were suggested to be a few degrees.[11,12] This means that a high-quality textured template must be developed on ceraflex before it can be used for the fabrication of HTS coated conductors. In the past few years, ion-beam assisted deposition (IBAD) has been successfully shown to obtain textured templates on non-crystalline metallic tapes for YBCO coated conductors. IBAD-MgO[13] can build good bi-axial texture in a few minutes.[14] The issue is that the texture quality in the IBAD-MgO textured template is sensitively dependent on the surface condition of the substrate, especially surface roughness.[15] The root-mean-square surface roughness R_{rms} ~ 3.5 nm[16] is generally regarded as the minimum requirement for a high-quality textured IBAD-MgO template to form. The typical surface roughness of commercial ceraflex is about 100 nm,[9] which is too rough and needs to be reduced by two orders of magnitude for the IBAD-MgO texturing process. IBAD-YSZ has been fabricated on ceraflex,[17] since it is more tolerant of the surface roughness, but the in-plane $\Delta\phi$ ~ 19° for YBCO on IBAD-YSZ textured ceraflex is not suitable for HTS coated conductors. In this paper, we report on the systematic study of ceraflex surface smoothing and IBAD-MgO texture development and show preliminary results of YBCO fabricated on textured ceraflex.

EXPERIMENTAL

Since the two kinds of ceraflex (MarkeTech International, Inc.) have the same surface condition, 8%Y ceraflex was chosen to examine the feasibility of the IBAD-MgO texturing process on ceraflex. The thickness was 0.1 mm, and typical dimensions were about 10 mm × 10 mm. Spin-on-glass (SOG) T14 (Honeywell Electronic Materials)[18] was coated on ceraflex to smooth the surface. SOG was spun on ceraflex at 2,000–4,500 rpm for 1 min and then baked at 400–600°C to remove the chemical elements. Multilayer SOG coatings were used to reduce the surface roughness further. The IBAD-MgO details have been introduced in another publication.[19] The Ar^+ beam parameters were 750 eV, 10 mA with 45° to the normal of sample stage. Before IBAD-MgO deposition, a 15–40 nm amorphous Y_2O_3 buffer layer was deposited by e-beam deposition and pre-exposed in Ar^+ for 1–2 min. A 9–11 nm MgO film was deposited by ion beam assisted e-beam deposition at room temperature with a growth rate of 0.15 nm/s. Subsequently, a 100–200 nm homo-epi MgO layer was grown at 0.05–0.1 nm/s and 300–500°C. A 150 nm $SrTiO_3$ film was deposited *ex situ* by pulsed laser deposition (PLD) at 690°C in 230 mTorr O_2. Preliminary YBCO growth was achieved after deposition of a 200–300 nm $LaMnO_3$ buffer layer by PLD at 750°C on textured ceraflex. The 300 nm YBCO film was grown by PLD at 780°C on this $LaMnO_3$ buffer. We used *in situ* real time reflection high energy electron deflection (RHEED) to monitor the texture development during IBAD-MgO and homo-epi MgO deposition. Surface morphology was examined by atomic force microscopy (AFM) and scanning electron microscopy (SEM). Film structure and epitaxy were measured by X-ray diffraction (XRD) θ–2θ and ϕ-scan. AC susceptibility measurement was used to examine the transition temperature (T_c) of the YBCO film.

RESULTS AND DISCUSSION

We first measured the original surface and structural properties of the ceraflex substrates, and the results are shown in Fig. 1. Figure 1a is an AFM image of the 8%Y ceraflex substrate with original R_{rms} = 105.8 nm, which is on the same order of the value provided by the manufacturer[9] for a 5 μm × 5 μm area. The grain dimension is about 1 μm, but gaps deeper than 100 nm always appear during this

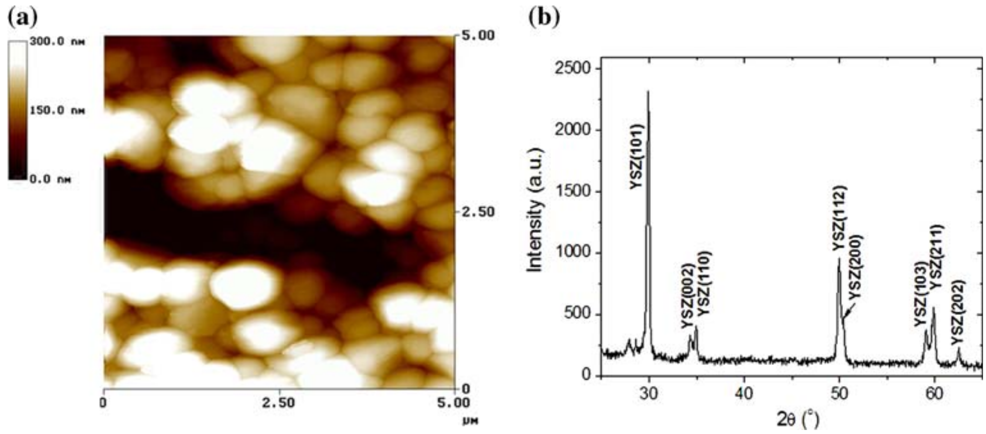

Fig. 1. Original surface and structure properties of 8%Y ceraflex. (a) The surface morphology measured by AFM, R_{rms} = 105.8 nm for a 5 μm $\times$ 5 μm area. (b) The XRD θ–2θ pattern.

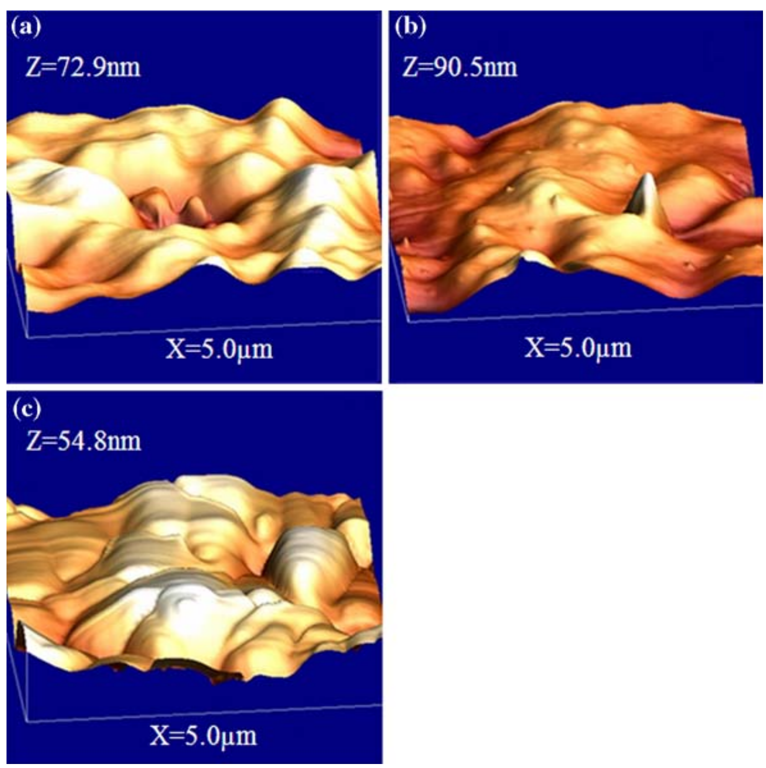

Fig. 2. Surface morphology of one layer SOG coated ceraflex, measured by AFM. (a) Spun at 2,000 rpm, R_{rms} = 12.1 nm; (b) spun at 3,000 rpm, R_{rms} = 11.0 nm; (c) spun at 4,000 rpm, R_{rms} = 8.2 nm. AFM scan area is 5 μm $\times$ 5 μm.

measurement. All the peaks in the XRD θ–2θ pattern in Fig. 1b belong to polycrystalline YSZ.[20] This confirms that the 8%Y ceraflex is pure untextured YSZ.

The deep gaps shown in Fig. 1a must be filled, and the surface needs to be smoothed for IBAD-MgO texturing. However, standard mechanical and chemical polishing processes are not suitable for our experiments, because of the mechanical properties, thickness and chemical stability of ceraflex. Since SOG has been widely used to smooth and planarize device surfaces in the semiconductor industry,[18] we chose SOG to reduce the surface roughness of ceraflex. One layer of SOG was spun on ceraflex and baked to fill the gap and reduce surface roughness. The AFM patterns of one-layer SOG coated ceraflex are shown in Fig. 2. In Fig. 2a, the SOG spun at 2,000 rpm has already filled the deep gaps shown in Fig. 1a, and the surface is changed to a fluctuant hill-like morphology. The R_{rms} is reduced to 12.1 nm for this 5 μm $\times$ 5 μm area. With increasing spin speed, the R_{rms} is continuously reduced to 11.0 nm and 8.2 nm, as shown in Fig. 2b and c, for 3,000 rpm and 4,000 rpm, respectively. These

improvements indicate that the higher spin speed results in smaller surface roughness. These results confirm that one layer SOG can fill the deep gaps on ceraflex and greatly reduce the surface roughness to less than 10 nm, but this is still not suitable for IBAD-MgO. Multiple SOG coatings might be able to reduce the surface roughness further. However, for the spin speed higher than 4,000 rpm, there are clear solidified SOG edges, which might have been caused by the surface tension during the spin process on ceraflex. These SOG edges would be accumulated and would damage the uniformity of the whole area for multiple SOG coatings. Therefore, the speed range from 3,000 rpm to 3,500 rpm was chosen for the following experiments.

The variation in the surface morphology of multiple SOG coatings is shown in Fig. 3 for a spin speed of 3,500 rpm. Figure 3a shows the one layer SOG coated surface, where gaps are filled and R_{rms} is reduced to 10.0 nm. After a coating of two layers SOG, the surface fluctuation becomes gentler, with R_{rms} = 5.1 nm, as shown in Fig. 3b. With three layer SOG the R_{rms} is further decreased to about 3.05 nm (Fig. 3c), which is already on the same order of IBAD-MgO requirement.[16] All the samples shown in Fig. 3a–c were baked immediately after each spin, but these multiple bakings decreased the efficiency of the process. To verify if one baking is feasible for multiple SOG coatings, we baked a two layer SOG coated sample only once before AFM examination. Figure 3d indicates that the surface becomes rougher, with R_{rms} = 11 nm, and more surface fluctuation is apparent than in Fig. 3b. This implies that baking immediately after each spin is necessary and that the first layer must be solidified before the next spin in order to reduce roughness. The above results demonstrate that three layer SOG coatings on ceraflex can provide sufficient surface smoothness for IBAD-MgO texturing. This improvement has been repeated on three layer SOG spun at 3,200 rpm, which has been used for the next IBAD-MgO texturing experiments, where R_{rms} ~ 3 nm. We estimated from the information provided by the manufacturer that the thickness of the three layer SOG spun at 3,200–3,500 rpm was less than 700 nm.

With amorphous Y_2O_3 buffer and a few minutes of pre-exposure in Ar^+, texture has been built by IBAD-MgO deposition on three layer SOG smoothed ceraflex. The texture development was monitored by *in situ* RHEED, shown in Fig. 4. The RHEED pattern in Fig. 4a demonstrates that the texture is built with 11 nm IBAD-MgO. And, with increasing homo-epi MgO thickness, texture quality is continuously improved; the clearer pattern in Fig. 4b confirms the greatly improved texture in the 100 nm homo-epi MgO layer.

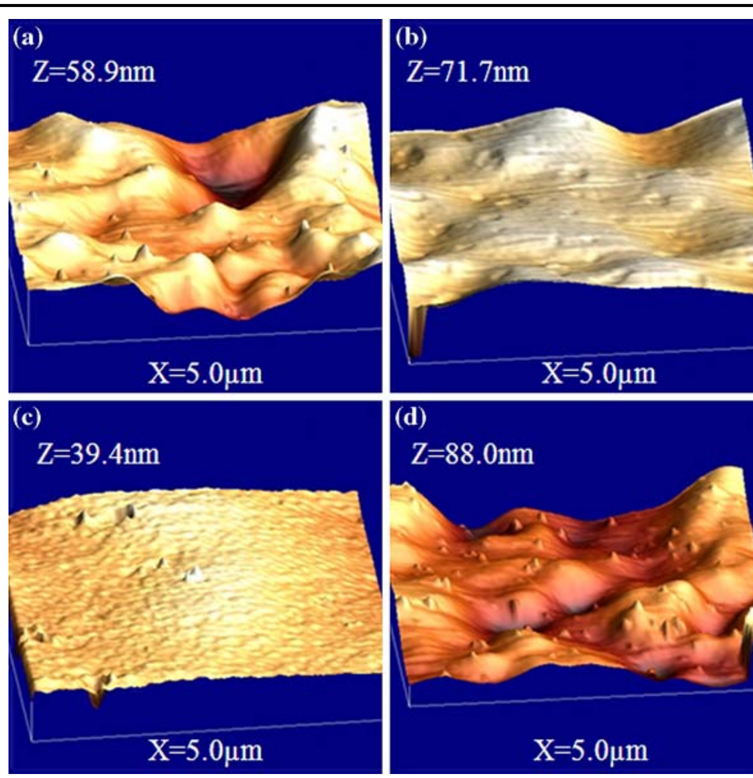

Fig. 3. Variation in surface morphology of multiple SOG coated ceraflex. SOG was spun at 3,500 rpm, 1 min for all the samples. (a) One layer SOG, R_{rms} = 10.0 nm; (b) two layer SOG, R_{rms} = 5.1 nm; (c) three layer SOG, R_{rms} = 3.05 nm. The samples in (a)–(c) were baked immediately after each spin. (d) Two layer SOG, baked only once, R_{rms} = 11.0 nm. AFM scan area is 5 μm × 5 μm.

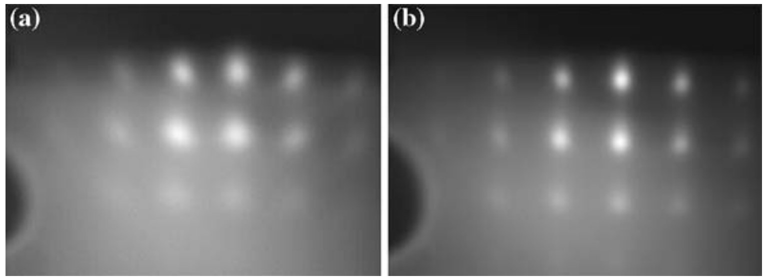

Fig. 4. *In situ* RHEED patterns during texture development of IBAD-MgO and homo-epi MgO deposition; (a) 11 nm IBAD-MgO, (b) homo-epi MgO(100 nm).

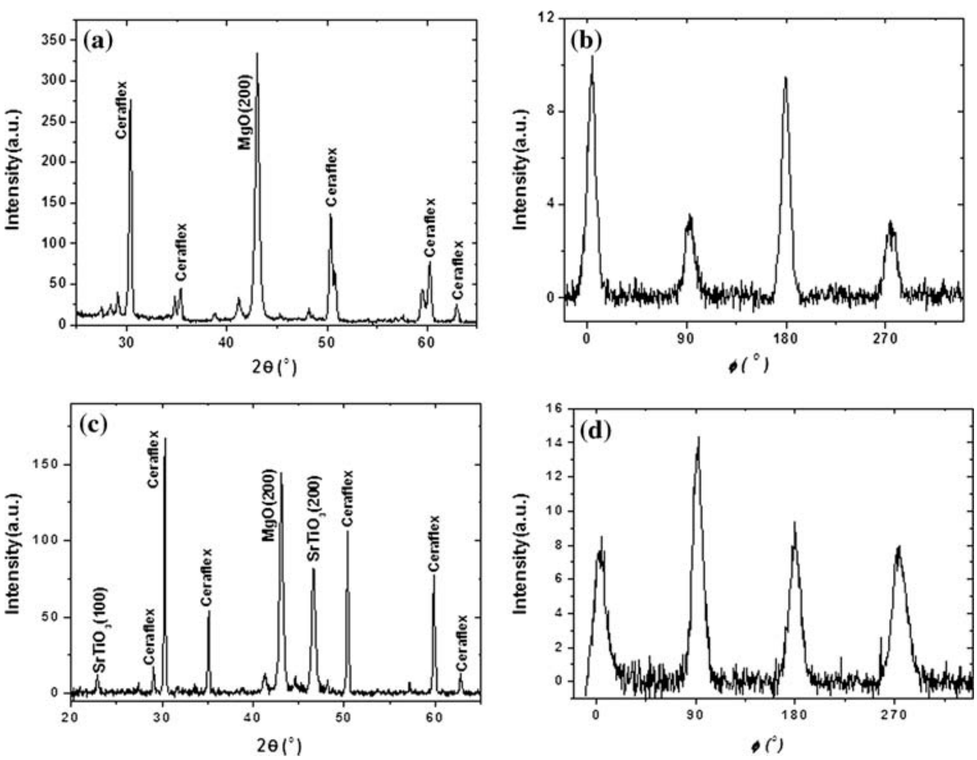

Fig. 5. Comparison of XRD patterns of homoepi-MgO(100 nm)/IBAD-MgO(11 nm)/Y_2O_3(15 nm)/SOG/ceraflex [(a) θ–2θ pattern, (b) MgO(110) ϕ-scan] and $SrTiO_3$ (150 nm)/homoepi-MgO(100 nm)/IBAD-MgO(9 nm)/Y_2O_3(15 nm)/SOG/ceraflex [(c) θ–2θ pattern, (d) $SrTiO_3$ (220) ϕ-scan.] Figure 5(c) is reused with permission from Rongtao Lu, Ronald N. Vallejo, Daniel W. Fisher, and July Z. Wu, Applied Physics Letters, 89, 132505 (2006). Copyright 2006, American Institute of Physics.

We deposited a 150 nm $SrTiO_3$ film to examine the feasibility of epitaxial growth of perovskite films on the textured ceraflex. Figure 5 compares the XRD patterns of the homo-epi MgO layer and the $SrTiO_3$ layer. The MgO(200) peak is very clear in Fig. 5a, and the out-of-plane full-width-at-half-maximum (FWHM) of the MgO(200) rocking curve is about 2.8° The MgO(220) ϕ-scan in Fig. 5b confirms that the in-plane $\Delta\phi$ is 9.3° The X-ray θ–2θ pattern in Fig. 5c indicates strong $SrTiO_3$(001) and (002) peaks, and the $SrTiO_3$(002) rocking curve proves that the out-of-plane FWHM is about 3° An $SrTiO_3$(110) ϕ-scan indicates that the in-plane $\Delta\phi = 10.8°$ Compared with homo-epi MgO, this higher in-plane $\Delta\phi$ might be induced by the unoptimized deposition conditions. These results demonstrate that the highly textured homo-MgO/IBAD-MgO template has been successfully fabricated on SOG smoothed ceraflex, and that epitaxial growth of perovskite films on this textured template is possible.

The surface morphologies of homo-epi MgO and $SrTiO_3$ are compared in Fig. 6. For homo-epi MgO, AFM (Fig. 6a) and SEM images (Fig. 6c) show that the surface is condensed and smooth, with R_{rms} = 3.89 nm and a grain size smaller than 0.5 μm. The images also show that the surface is very clean over a large area. For the $SrTiO_3$ film (Fig. 6b and d), the grains become much bigger, due to the high temperature growth. The surface roughness is

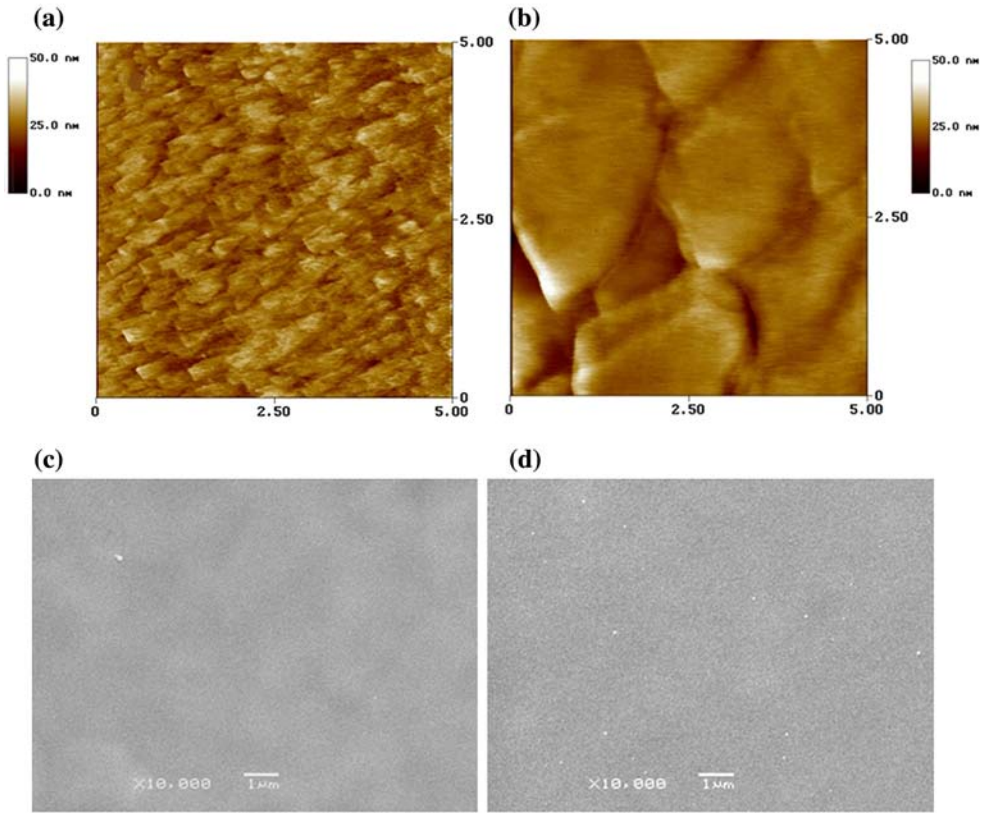

Fig. 6. AFM [(a)–(b)] and SEM [(c)–(d)] images of homo-MgO/IBAD-MgO/Y_2O_3/SOG/ceraflex [(a) AFM image, R_{rms} = 3.89 nm; (c) SEM image] and $SrTiO_3$/homo-MgO/IBAD-MgO/Y_2O_3/SOG/ceraflex [(b) AFM image, R_{rms} = 4.08 nm; (d) SEM image].

R_{rms} = 4.08 nm, and the surface is still very uniform over a large area. As a typical ABO_3 perovskite material, $SrTiO_3$ has been widely used as a buffer layer for the growth of many perovskite films;[1,4] this smooth, uniform and clean surface makes epitaxial growth of other perovskite films feasible.

In the preliminary YBCO deposition experiments, $LaMnO_3$ was deposited as a buffer layer. The structure and superconducting properties are shown in Fig. 7, and more detailed results will be reported later.[21] The XRD θ–2θ pattern (Fig. 7a) confirms that the phase structure is very pure, since the YBCO (00l) peaks are primary and all other peaks belong to the epitaxial buffer, textured template and polycrystalline substrate. The YBCO (110) ϕ-scan indicates that the in-plane $\Delta\phi$ ~ 6.8° AC susceptibility measurements are given in Fig. 7b and determine the T_c to be about 88 K, although the transition is broader than desired. Even so, these initial results confirm that epitaxial YBCO superconducting films can be successfully fabricated on buffered and textured ceraflex; better performance can be expected with optimized conditions, which are underway. SOG induced Si diffusion is a possible problem. The baking of SOG at higher temperature and the use of a barrier layer between SOG

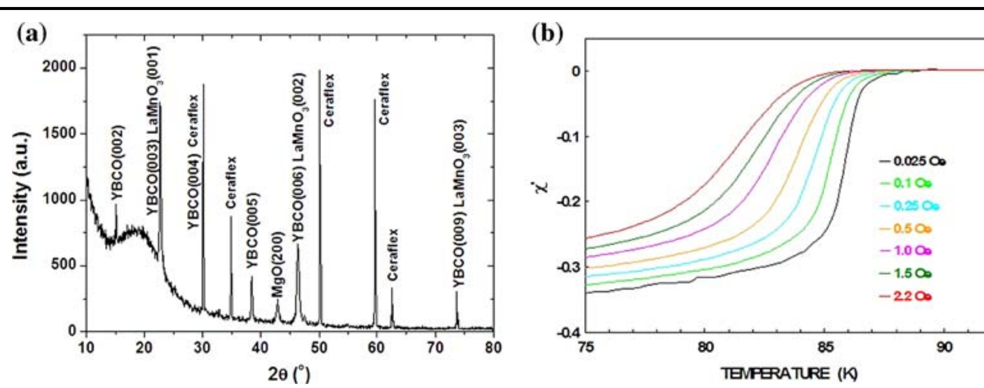

Fig. 7. Properties of YBCO/$LaMnO_3$/homo-MgO/IBAD-MgO/Y_2O_3/SOG/ceraflex. (a) XRD θ–2θ pattern. (b) AC susceptibility curve.

and buffer layers will be explored to eliminate this diffusion. A potential concern with the use of ceraflex substrates is that, after processing, the processing heat tends to make the substrates more brittle, but further investigation of this is required for verification.

CONCLUSION

The systematic study of textured IBAD-MgO templates on non-metallic flexible ceraflex, and the feasibility to grow epitaxial perovskite films on top, has been demonstrated in this work. The original rough surface of ceraflex was smoothed by multilayer SOG, and the surface roughness was reduced to a few nanometers, which is qualified for the IBAD-MgO texturing process. Bi-axial texture was built with 9–11 nm IBAD-MgO, and the best inplane $\Delta\phi \sim 9°$ was obtained on 100–200 nm homoepi MgO film. Different epitaxial perovskite films were fabricated on the textured template. Multilayer epitaxial $SrTiO_3$(150 nm)/homo-MgO(100 nm)/IBAD-MgO(9 nm)/Y_2O_3(15 nm)/SOG/ceraflex was fabricated with in-plane $\Delta\phi \sim 10.8°$ Multilayer epitaxial YBCO(300 nm)/$LaMnO_3$(200 nm)/homo-MgO(100 nm)/IBAD-MgO(9 nm)/Y_2O_3(15 nm)/SOG/ceraflex was successfully demonstrated with pure c-axis orientation structure and representative superconductivity. Optimization of the whole process is still in progress.

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